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Investigations on the Effect of Selected Post-Treatment with Thermal and Oxidizing Agents on the Characteristics and Performance of Polysulfone Hemodialysis Hollow Fiber Membranes

Aya N. Mohamed [a,b,*], Heba A. Hani [a], Shadia R. Tewfik [a], Reem S. Ettouney[b], Mahmoud A. El-Rifai [b]

[a]*Chemical Engineering and Pilot Plant Department, National Research Centre, Dokki, Giza, Egypt*

[b]*Chemical Engineering Department, Cairo University, Cairo, Egypt*

Abstract

Hemodialysis plays a crucial role in the lives of patients with kidney failure. This study explores the effects of post-treatment using heat and oxidizing agents on the characteristics and performance of polysulfone (PS) hemodialysis hollow fiber membranes. The PS hemodialysis hollow fiber membranes were produced using the dry-wet spinning technique and subsequently post-treated to enhance their performance and surface features. The post-treatment was performed alternatively using heat, and two oxidizing agents, namely hydrogen peroxide (H_2O_2) and sodium hypochlorite (NaOCl). The membranes were characterized using various techniques, including scanning electron microscopy (SEM), measurement of water contact angle, surface roughness analysis, average porosity assessment, pore size evaluation, and mechanical properties testing. The performance of the prepared hollow fiber membranes was also examined. The results showed that the post-treatment with H_2O_2 significantly improved the membranes' mechanical properties, where break strength increased by 7.4% and 9%, while break strain increased by 55.5%and 29% for heat treatment and H_2O_2 treatment respectively, with only minimal effects on the membrane's morphology and porosity. Additionally, the ultrafiltration coefficient decreased from 21.9 to 11.2 L m^{-2} h^{-1} bar^{-1}, while the sieving coefficients for urea and creatinine approached unity for both treated and untreated membranes. In contrast, post-treatment with NaOCl led to significant deterioration of the membrane's mechanical properties and structure under the conditions studied. These findings suggest that H_2O_2 post-treatment is a promising strategy for enhancing the mechanical properties and tuning the ultrafiltration coefficient of PS-based hemodialysis membranes, without compromising their overall performance.

Keywords: Hollow fiber Membrane; Polysulfone; Hemodialysis; Oxidizing agent; Post-Treatment.

1. Introduction

Hemodialysis is of vital importance for chronic kidney disease patients. Dialysis membranes are used to remove accumulated uremic toxins, excess water and ions in addition to providing insufficient ions to the patient's blood. The hemodialysis hollow fiber (HDHF) membrane should be of high selectivity, blood compatibility, and fouling resistance [1]. PSis one of the most widely used polymers for HDHF membranes due to its excellent chemical and physical properties [2–4]. However, undesirable interactions with blood, such as protein adsorption, complement activation, and surface-induced coagulation, still occur upon contact with blood[5,6]. Currently, researchers are intensifying their efforts to improve the hemocompatibility of dialysis membranes while maintaining acceptable separation performance.

Ultrafiltration rate and separation performance of HDHF membrane mainly depend on the mean pore size, average porosity and membrane thickness [4]. The hemocompatibility of a HDHF membrane is directly related to its surface properties such as roughness, charge, and hydrophilicity [7,8]. Hydrophilic membranes have increased biocompatibility as hydrophilic modifications of dialysis membranes reduce protein adsorption; however, highly hydrophilic surfaces result in elevated complement, leukocyte activation/leukopenia [4,9,10]. The roughness of PSmembranes is associated with albumin adsorption, thus, dialysis membranes with a smoother surface have lower protein adsorption [11]. HDHF membranes need to be negatively charged to prevent the adhesion of negatively charged blood proteins and to avoid blood coagulation.

*Corresponding author: ayanaguib17@gmail.com; (**Aya Naguib Mohamed**)
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Negatively charged HDHF membranes with a near-neutral zeta potential have reduced blood interaction and complement activation [9,12]. There are several methods for improving HDHF membranes hemocompatibility including polymer blending in addition to physical and chemical post-treatment [2,7].

Treatment with oxidizing agents controlsthe membrane surface properties and pore size. Oxidants such as NaOCl and H_2O_2 are used for cleaning fouled polymeric membranes. NaOCl is a low-cost oxidant while H_2O_2 is a green environmentally friendly oxidizer that only releases water as a by-product [13–15]. Potassium persulfate is a strong stable oxidizing agent and could be used as an alternative to chlorine-based disinfecting agents [16]. A comprehensive study investigated the effect of H_2O_2 on PS membrane treatment. The effect of treatment time, temperature and concentration on membrane contact angle, porosity and flux were investigated [17]. This study revealed that H_2O_2 treatment has an apparent effect on membrane surface properties and separation performance and that at reduced temperature and concentration membrane degradation can be avoided. Other studies showed that long time exposure to oxidizing agents could result in apparent cracks on membrane surface and deterioration in mechanical properties [18,19]. Few studies focused on treatment with oxidizing agents of hemodialysis membrane. Wolff and Zydney [20], studied the effect of hypochlorite solution on commercial Fresenius PS hollow fiber membrane pore size and they stated that it is possible to control membrane pore size by controlling time, temperature and concentration of hypochlorite solution. Significant changes occurred in the commercial Fresenius PS HDHF membrane morphology and CA after NaOCl treatment [14].

To the best of the authors' knowledge, this study is the first to investigate and compare the effects of H_2O_2 and NaOCl as oxidizing agents along with thermal treatment, on PS membranes for hemodialysis applications under defined conditions. The main objective of this study is to compare alternativepost-treatmenttechniques using heat andoxidizing agents,namelyH_2O_2 and NaOClforPS HDHF membrane. The effect of these post-treatment techniques on the membrane characteristics and performance was thoroughly evaluated.

2. Materials and Methods

2.1. Materials

PS, Ultrason S6020, Mw=62,000 g $mole^{-1}$,(M_n/M_w=3.9) purchased from (BASF, Germany), was used as the base polymer for the HF. Polyethylene glycol (PEG) having a molecular weight of 35000 g $mole^{-1}$ was used as a pore forming agent and was purchased from Merck(water $\leq$ 1% and sulfated ash$\leq$ 0.2 %). N-methyl-2-pyrrolidone (NMP), supplied by Carl-Roth (>99.8%), was used as a solvent for both dope and pore fluids. Glycerol 99% and Formalin (37%) were used as preservative solutions and were supplied from Fisher Chemicals, Germany and PIOCHEM laboratory chemicals, Egypt, respectively.H_2O_2 9% and NaOCl, available chlorine 12-14%, were purchased from Luna Egypt and Alpha Chemika India, respectively.Sodium hydroxide pellets 99% and hydrochloric acid 30-34% for pH adjustment during post-treatment were purchased from Modern Lab Laboratory Chemicals, Egypt and El-Nasr Company for Pharmaceutical Chemicals, Egypt respectively. Urea and creatinine used for membrane performance evaluation were purchased from El-Nasr Pharmaceutical Chemicals Co. and Alpha Chemicals Co., respectively.

2.2. Membrane preparation

PSHDHF membranes were prepared using the dry-wet phase inversion method[21]. The membrane spinning system is schematically represented in **Figure 1**. The dope solution was prepared by gradually adding 18 wt. % PS and 6 wt. %PEG into NMP solvent at 70°C in a jacketed mixing tank. Stirring continued for 2 days at 400-500 rpm under nitrogen pressure (2-3 bars) to obtain a homogeneous dope solution. Degassing was carried out using a vacuum pump to remove air bubbles from the polymer solution. The fibers were spun by coextrusion of the dope solution, and a mixture of water and NMP as bore fluids in the lumen side through a spinneret with an inner diameter of 280 μm and an outer diameter of 1 mm. Nascent fibers were immersed in a coagulation bath containing water as a non-solvent to remove solvent and solidify fibers at room temperature. Fibers were then rinsed in a water bath for thorough washing to remove any residual solvent. Spun fibers were then collected on a reel winder.

The produced HFs were soaked in RO (reverse osmosis) water until the next day. Washed membranes were stored in a solution of 10% glycerol and 1% formalin until further analysis and post-treatment. HF spinning conditions are summarized in **Table 1**.

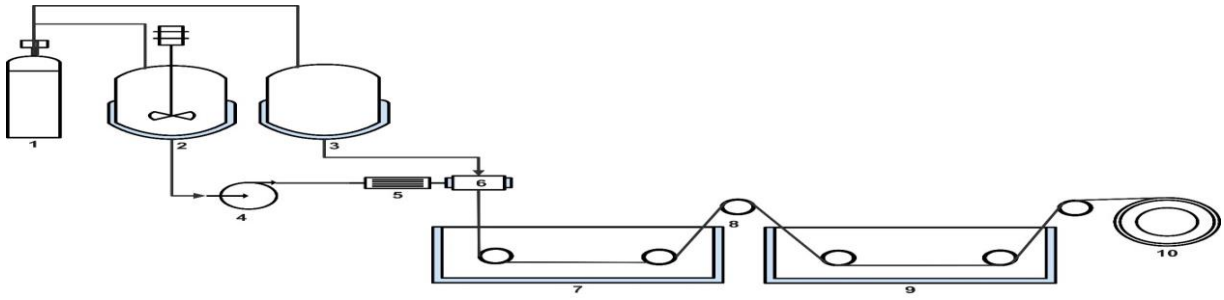


Figure 1: HDHF membrane spinning system, 1: Nitrogen cylinder, 2: Jacketed dope tank equipped with agitator, 3: Jacketed bore fluid tank, 4: Pump, 5: microfilter, 6: Spin block, 7: Coagulation bath, 8: Rollers, 9: Washing water bath, 10: Winder

Table 1: PSHDHF membrane spinning conditions

Spinning parameter	Value
Ratio (Dope to bore flow)	1.5
Dope solution temperature (°C)	70
Bore fluid temperature (°C)	60
Spin block temperature (°C)	35
Air gap (cm)	25
Coagulation bath solution	RO water
Coagulation bath temperature (°C)	22

2.3. Membrane post-treatment

By reviewing and analysis of numerous proposed HF membrane treatment we came up with preferred conditions [17–20,22–26]. PS HF samples (stored in 10% glycerol and 1 % formalin) were washed several times in RO water at room temperature, then soaked in RO water until the next day to remove any traces of preservative solution.The pristine samples were then alternatively post treated as follows:

a) Heat treatment:samples were soaked in 500 mL RO water; pH was adjusted at 8 using NaOH (7%)
b) H_2O_2 treatment: samples were soaked in 500 mL solution of 7500 ppm H_2O_2; pH was adjusted at 8 using 7% NaOH.
c) NaOCl treatment: samples were soaked in a500 mL solutionof 7500 ppm NaOCl, pH was adjusted using 10% HCl.

Post-treatment experiments were performed for 3 hours at 45°C in a closed water bath placed in a fuming cupboard. Post-treated fibers were taken out, left to cool, then washed and soaked in RO water until the next day for characterization. Pristine, heat treated, H_2O_2 and NaOCl treated samples were coded as A, AC3, AH and AN, respectively.

2.4. Membrane characterization

2.4.1. Proton nuclear magnetic resonance (H-NMR) analysis

H-NMR spectra of the membrane samples were obtained using JEOL ECA NMR spectrometer using DMSO as the solvent.

2.4.2. Fourier transform infrared (FTIR) analysis

The changes in functional groups of the membrane were analyzed using FTIR analyzer. FTIR analysis was performed using infrared spectrophotometer (FT/IR-6100) from A Jasco, Japan detector with the transmittance mode. All spectrums covered a range from 400 cm^{-1} to 4000 cm^{-1}.

2.4.3. SEM imaging

The morphology of pristine and post-treated PS HF was studied using bench-top SEM(JEOL SEM 6000 Neoscope desktop). Fibers were washed several times and soaked in RO water for a day, then air dried. HF samples were cut using a sharp razor, and gold sputtered for 30 seconds before SEM imaging. Also, elemental analysis was performed for fiber surface using energy dispersion spectroscopy (EDS).

2.4.4. Roughness

The surface roughness of the membranes was analyzed using a TT-AFM workshop with a resolution of 1.5 μm. The HF samples were fixed using double-sided tape on a magnetic plate[27]. The vibrating scanning mode was used for a scanning area of 5 μm × 5 μm on the outer membrane surface. Roughness variables were analyzed using Gwidyyon software.

2.4.5. Water contact angle (CA)

The water CA of the HF membranes was measured using OCA 15EC contact angle instrument (Data Physics Instrument) through a digital video image of the water drop on the dried outer surface of the HF at 25°C. For each sample, the CA was measured at five different positions from which the average CA and standard deviation were estimated.

2.4.6. Mechanical properties

The mechanical properties of pristine and post-treated PS HF were studied using a benchtop Tinius Olsen H5kS universal tensile testing machine connected to 5N load cell and tested fiber length of 10 cm. Horizon software package was used to estimate membrane tensile stress, break strain and Young's modulus.

2.4.7. Average porosity (ε)

The average porosity of the HF membranes was measured using the gravimetric method [28], where the weight of kerosene entrapped within the membrane pores was measured and porosity was calculated from equation 1.

$$\varepsilon(\%) = \frac{\frac{W_W - W_D}{D_{kerosene}}}{\frac{W_W - W_D}{D_{kerosene}} + \frac{W_D}{D_{Polymer}}} * 100 \qquad (1)$$

Where ε is the membrane porosity (volume %), W_W is the weight of the wet membrane (g), W_D is the weight of the dry membrane (g), $D_{kerosene}$ the density of kerosene (0.82 g cm^{-3}), $D_{Polymer}$ is the density of PS (1.24 g cm^{-3}).

2.4.8. Mean pore size and pore size distribution

Mean pore size and pore size distribution for the prepared PS HF membranes were determined using Belsorp Max apparatus (MicrotracBel. Corp.) [29].

2.5. Membrane performance evaluation

Membrane performance was assessed in terms of pure water permeability (ultrafiltration coefficient) and sieving coefficient for small molecules. Mini modules were prepared for this purpose. A bundle of 20 fibers was potted in a glass module using epoxy resin and hardener where the effective length of the fibers was 20 cm. Experiments were conducted on HYFLUX membrane evaluation device using in-out mode as shown in **Figure 2**.

2.5.1. Permeability

Pure water permeability was determined from the slope of a linear fit between the water flux and transmembrane pressure according to equation 2

$$L_p = \frac{J_s}{\Delta P} \qquad (2)$$

Where L_p is the pure water permeability (L $m^{-2}h^{-1}bar^{-1}$) and Δp is the transmembrane pressure (bar).
The pure water flux was calculated using equation 3

$$J_s = \frac{V}{A\Delta t} \qquad (3)$$

Where J_s is the water flux (L $m^{-2}h^{-1}$), V is the permeate volume (L), A is the effective membrane area (m^2), Δ t is the permeate collection time (h).

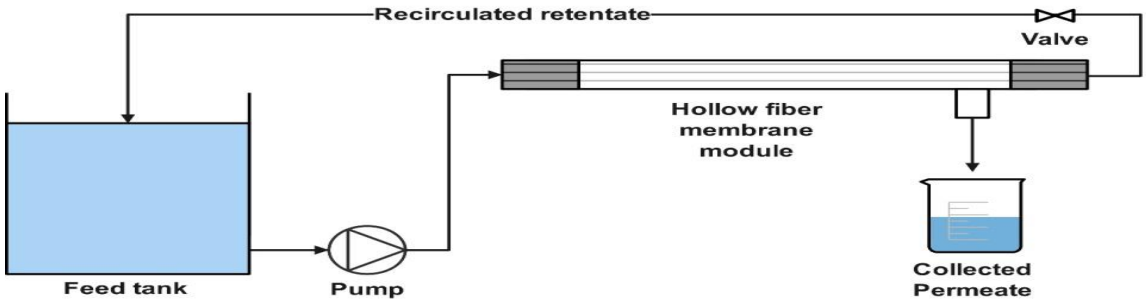


Figure 2: Membrane performance evaluation experimental system setup

2.5.2. Sieving coefficient (SC)

Membrane separation performance was assessed using the experimental system shown in **Figure 2**. Urea 99% (MW 62 Da) and creatinine 99% (MW 113.12 Da) were used representing small toxins in the blood. All experiments were performed under constant pressure (TMP 0.5 bar) at room temperature. Test solutions (5L) were prepared at different concentrations: for urea ranging between 300 to 1000 ppm while for creatinine the range was (60-150) ppm. All solutions were prepared in 0.9% sodium chloride saline solution. Permeates were collected after 30 minutes from start of operation, to ensure stability, then collected after 1, 2 and 3 hours to study the effect of time on the sieving coefficient. The concentrations of urea and creatinine were analyzed using UV-Vis spectrophotometry. The sieving coefficient was calculated from equation 4.

$$SC = \frac{C_p}{C_f} \quad (4)$$

Where SC is the sieving coefficient, C_f and C_p are the feed and permeate concentrations respectively.

3. Results and Discussion

3.1. Membrane structural analysis

The FTIR and H-NMR of the pristine and post-treated PS HF membranes are presented in **Figure 3**. The characteristic transmittance bands in the spectrum of PS are at 1148.81 cm^{-1}, 1238.19 cm^{-1} and 1583.73 cm^{-1} representing (O-S-O stretching), (CO-C stretching), and (C-C Aromatics), respectively [30,31]. These characteristic bands were observed in all membrane samples indicating no change in the membrane structure. Characteristic peaks intensity was lower for NaOCl treated samples. Other studies [19] also observed significant decrease in absorbance value after hypochlorite post-treatment of PES membrane at pH 9 and 12, which was attributed to the increase in the membrane surface roughness and depletion of the main polymer on the surface. Conversely, peak intensity was higher for H_2O_2 treated samples indicating membrane stability.

The intensity of the peak at 1486 cm^{-1} representing C–S vibration was lower in case of NaOCl treated PS membrane compared to pristine and H_2O_2 treated membranes, which was also observed in another study [22] for PES UF membranes treated using hypochlorite indicating a weaker C–S bond. While the band at 3650–3450 cm^{-1} represents the hydroxyl group[32,33] and indicates the presence of PEG, which appeared in all samples with low intensity for samples AH and AN, which indicate that the used oxidizing agents cause PEG degradation.

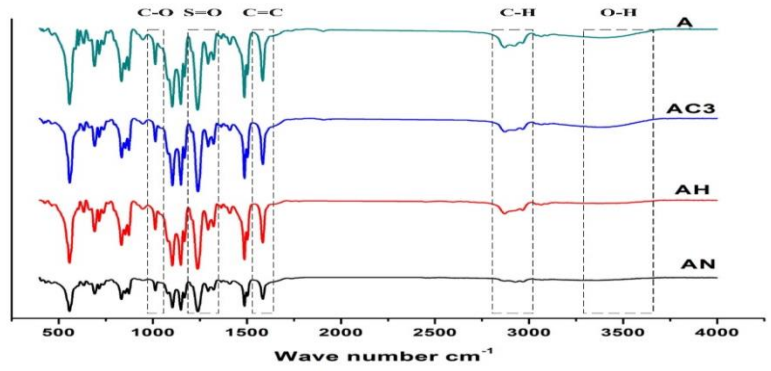
The change of overall membrane properties is affected by the additive fate after treatment [34]. A slight decrease in the IR absorbance of the additive (after exposure to NaOCl solution, as reported by Susanto H et al. 2009 [35] , indicates instability of PEG additive on PES membrane matrix. Other study demonstrated oxidation of the hydrophilic additives after exposure toNaOCl[36]. Several studies demonstrated that NaOCl is highly reactive with polyvinylpyrrolidone (PVP), where reaction rate goes to maximum at neutral to slightly basic pH [34,37]. In a previous study, anew beak appeared at 1735 indicating presence of C=O group confirming partial degradation of primary alcohol oxidation (PVP) for PS membrane treated with NaOCl at pH=9 [38].

H-NMR analysis also confirms PS membrane structure stability, as no change occurred for the main characteristic bonds of PS [39,40]. The peak at 5 ppm represents the hydroxyl group and indicates the presence of PEG, which disappeared in samples AH and AN indicating that the oxidizing agent caused PEG partial elution from the membrane.

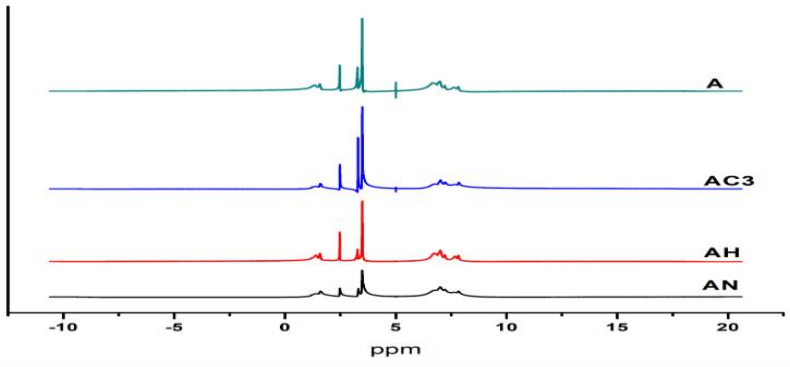
The elemental analysis of the main elements and traces present on the membrane surface from EDS data are shown in **Table 2**.The results show almost similar C, O and S percentages for A, AC3 and AH samples, which ensure stability of the membrane structure, while sample AN showed lower C and O percentages. Elemental analysis of hypochlorite treated membranes shows traces of sodium and chloride. Yadav et al. [19] also observed presence of traces of chlorine on PES membrane surface treated at pH 9, while no traces of chlorine were observed at pH 12. Rouaix [18] found chlorine traces in the elemental analysis after PS membrane treatment with 400 ppm hypochlorite solution at pH 8 after one day of post-treatment.

Table 2: Elemental analysis of pristine and post-treated membranes

Sample code	C	O	S	Na	Cl
A	64.44	29.37	6.19	0	0
AC3	65.49	28.51	6.01	0	0
AH	67.05	27.21	5.73	0	0
AN	62.95	26.825	2.425	4.525	3.275



A



B

Figure 3: Membranes structure analysis (A) FTIR spectra 4000–400 cm^{-1}, (B) H-NMR analysis

3.2. HF morphology

3.2.1. SEM images

SEM images shown in **Figure 4**were taken to study the morphology of pristine, heat, H_2O_2 and NaOCl post-treated membranes. The cross section of the pristine membrane shows that it possesses a double-layered structure with the inner layer responsible for separation and an outer finger-like structure which improves water permeability. The presence of NMP in the bore fluid helps in forming a spongy structure near the inner surface which increases membrane permeation. A significant decrease in the thickness of the outer active layer occurred only in the case of NaOCl treated sample. These results indicate that treatment using hypochlorite can be used to control active layer thickness leading to a decrease in the flow resistance and increase in the membrane flux. Defects and folds appeared on the outer surface of hypochlorite treated membranes, while no defects were observed in case of H_2O_2 and heat-treated (AC3) samples. Yadav et al. [19] also observed the formation of cracks on Koch PES UF membrane after soaking in 700 ppm hypochlorite solution for an exposure time of 15-35 days at pH 9 at 55ºC, while no defects were observed for membranes treated at a pH 12. NaOCl dissociation and oxidation potential are strongly dependent on pH [18,19,41]. Hypochlorous acid (HClO) is responsible for membrane degradation which decreases with increasing pH over alkaline range. The dimensions of pristine and post-treated fibers are presented in **Figure 5**. A slight decrease in the membrane's inner and outer diameters was observed for heated or H_2O_2 treated fibers, while the NaOCl sample showed the lowest fiber thickness among the treated samples. These results agree with Kai Li [22] who reported that H_2O_2 has a lower destructive effect on the PES membrane as compared to NaOCl. The degradation of the membrane, which is treated with H_2O_2, can be reduced by reducing the concentration and temperature of this oxidizing agent [17].

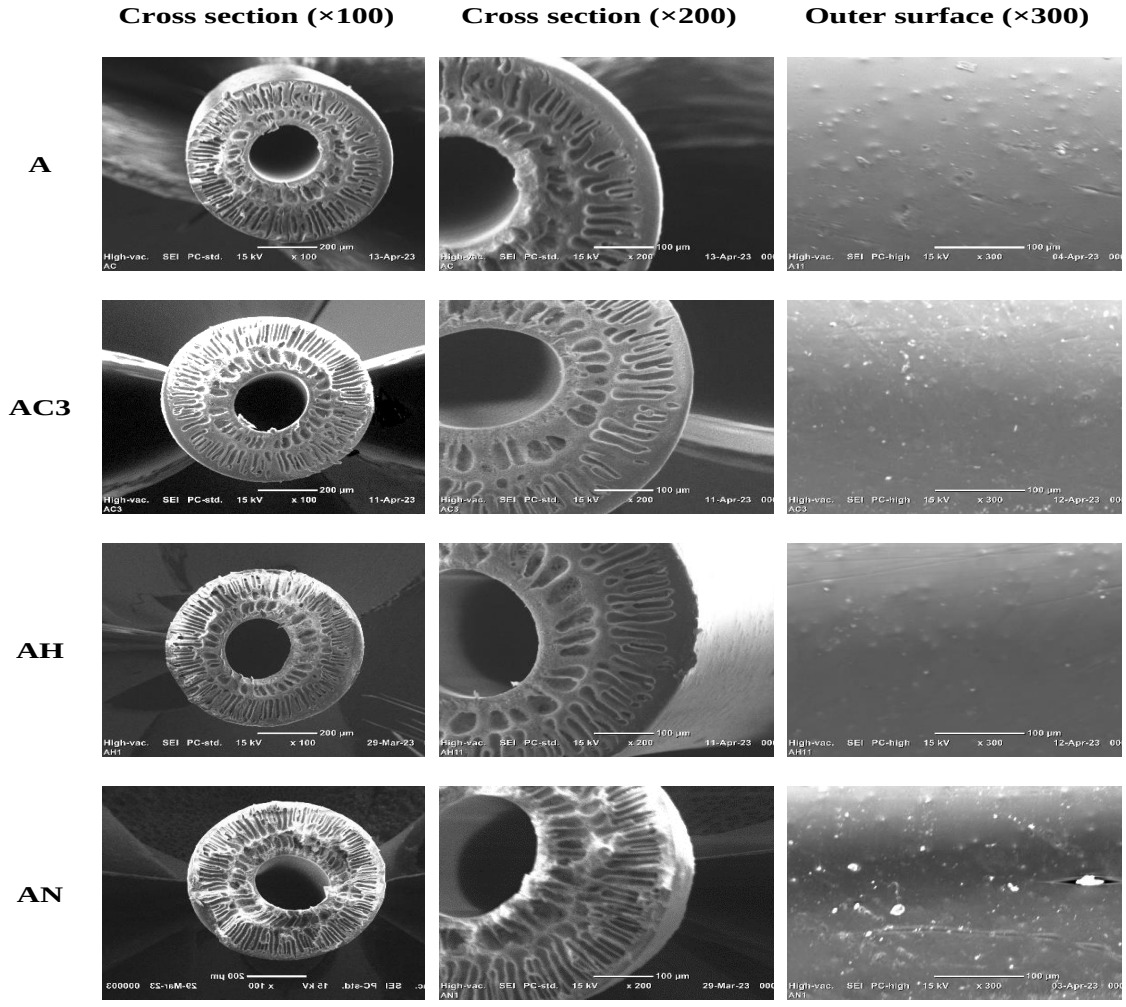


Figure 4: SEM images of pristine and post-treated HF membranes

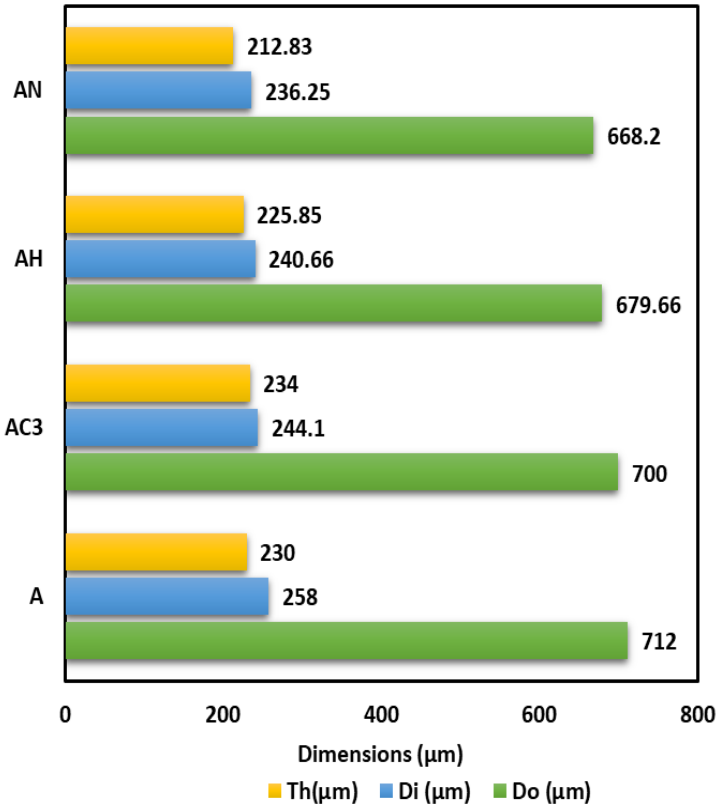


Figure 5:Dimensions of studied pristine and post-treated membrane fibers

3.2.2. Membrane roughness: AFM

AFM analysis (**Figures 6 and 7**) revealed a noticeable increase in membrane surface roughness after all post-treatment methods, with the most significant change observed in membranes treated with NaOCl. As shown in the 3D topographi, NaOCl-treated membranes exhibited a rougher and more irregular surface compared to both the untreated and hot water-treated samples. This increase in roughness is attributed to oxidative degradation of the main polymer and the removal of hydrophilic additives (PEG), resulting in pore enlargement and surface erosion.

The hot water treatment also increased surface roughness but to a lesser extent. These findings are consistent with previous work by Saghafi et al. [42], who reported an increase in PS/PVP membrane roughness from 9.2 to 21.3 nm after NaOCl treatment, and by Abdel-Karim et al. [43], who observed similar surface changes in PES membranes. While the rougher surface may enhance hydrophilicity and water flux, it could also promote fouling due to the larger surface area and more available adhesion sites for contaminants.

Surface roughness plays a major role in HD membrane hemocompatibility. Increased roughness increases the shear stress of the blood components near the membrane surface, which can lead to hemolysis or rupture of red blood cells (RBC) [44]. In addition, RBC rupture can enhance protein adsorption and platelet adhesion, leading to coagulation pathways and thrombus formation [45].

Westphalen et al. [45] measured the surface roughness of commercially available CTA and PAES HD membranes, where the Ra values were (5.4,7.5) and (10.5,15.9) nm for inner and outer surfaces, respectively. The results obtained for heat and H_2O_2treatment agreed with the reported roughness for commercial membranes.

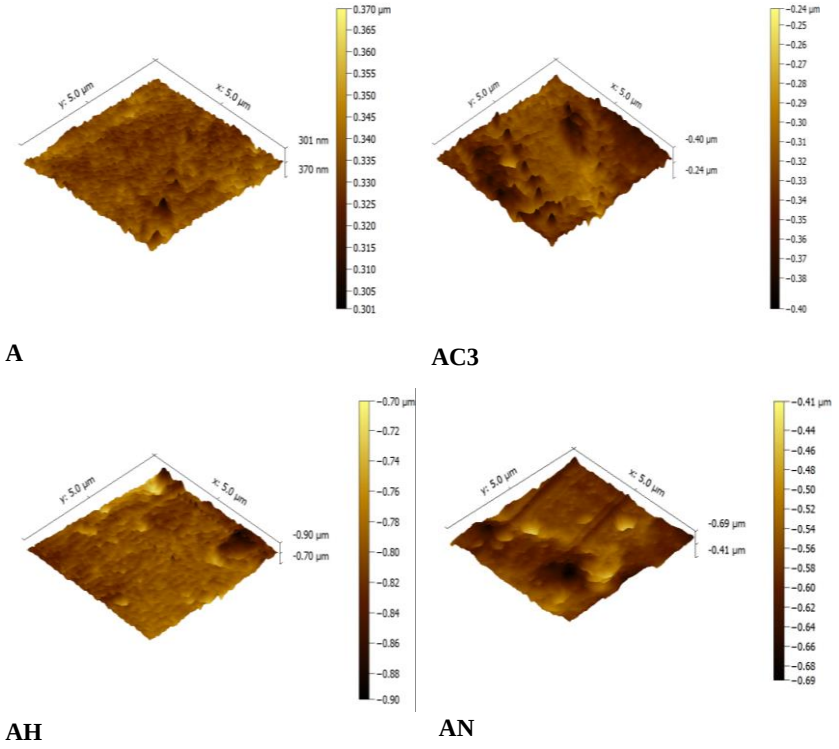


Figure 6: AFM images of studied HDHF membranes

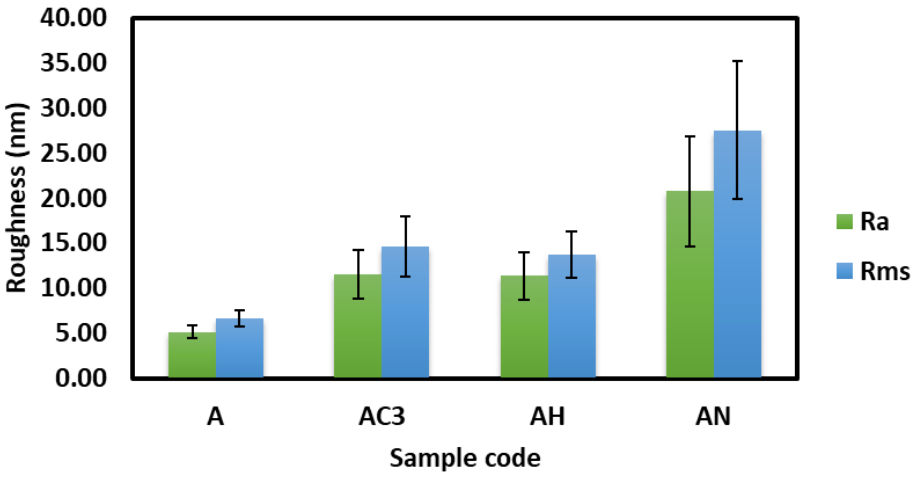


Figure 7: Pristine and post-treated PS HF membranes roughness

3.2.3 Membrane hydrophilicity

The CA of pristine and post-treated membranes is shown in **Figure 8**, where the error bars represent the standard deviation. Results indicated stability in the membrane CA upon treatment. Average CA values were 86.8°, 82.43°, 85.42° and 86.2° for pristine, heat treated, H_2O_2 treated and NaOCl samples, respectively. According to Özay et al. [17] increasing H_2O_2 treatment temperature from 80°C to 100°Cresulted in a slight increase in the CA from 59.51° to 65.80° for PES UF

membrane at constant concentration and time (1 mM and 55 min). Another study showed that the PES/PVP membrane CA increased after treatment with H_2O_2 due to the removal of some PVP from the membrane surface [13]. This study showed that although NaOCl treatment led to a decrease in the water CA and an increase in the negative charge of the surface, the fouling-propensity was increased. Zhang et al. [38] indicated that the water CA of NaOCl treated PS membranes decreased from 74º to 60º which was attributed to the chain scission of C-S bond. However, Dibrov et al.[23] revealed that PS/PVP UF membrane CA increased from 50º to 90º after treatment using 5000 ppm hypochlorite solution corresponding to a pH 11.5 for 4 hours at room temperature due to PVP degradation and wash out. Arkhangelsky et al. [30] found that the CA of PES membrane treated with NaOCl decreased despite the presence of PVP in the membrane dope solution. This study suggested two possible reasons, one is the formation of a charged phenyl sulfonate group that is more hydrophilic, and the other is the increased pore size on membrane surface which may be responsible for spreading of droplets by capillaries. The CA depends on several factors, including the loss of the hydrophilic additive, the increase in pore size, and the increase in surface negativity due to the degradation of the main polymer and additive, which does not necessarily mean less susceptibility to fouling.

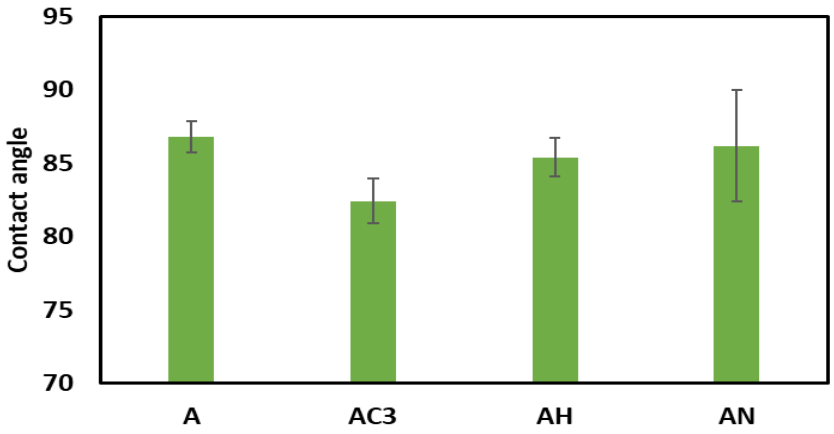


Figure 8:CA of pristine, heat, and oxidizing agents post-treated PS HF membranes

3.3. Membrane mechanical properties

The effect of heat and oxidizing agentspost-treatment on membrane mechanical properties is presented in **Table 3**. Interestingly, heat treated samples showed higher break strain and break stress compared to the pristine sample, indicating that heat treatment at selected conditions improves the mechanical properties of HF membranes. This result agrees with a previous result on electro spun PS membrane which revealed that membrane mechanical properties improved by increasing treatment temperature and time up to 190°C [46]. Also, H_2O_2 treatment increased membrane break stress and strain, compared to the pristine sample. The effect of H_2O_2 on the PS membranes' mechanical properties has not been previously reported. However, a study on PVDF membranes concluded that no deterioration occurred after treatment using 2-10 mg/l H_2O_2 solution [47].

Break strain and break stress dramatically decreased after hypochlorite treatment at selected conditions. Moreover, larger values of standard deviation were observed for the hypochlorite-treated samples indicatingnon-uniform fibers under the studied conditions. Arkhangelsky et al.[30] reported deterioration of the mechanical properties of PES membrane after exposure to hypochlorite 50 g h/L of free chlorine. Causserand et al. [48] revealed PS membrane degradation when treated using NaOCl solution at pH 8 and 10 owing to higher HClO concentration. Rouaix et al. [18], also observed significant decrease of both strain and tensile strength of PS membrane after 4 days soaking in NaOCl solutions (100ppm) under slight alkaline conditions. The effect of hypochlorite solution pH on membrane mechanical properties was also studied and it was concluded that at pH 8, the most significant decrease in membrane strain was observed [34]. Yadav et al. [19] observed more severity in mechanical properties deterioration of PES membrane at a pH of 9 compared to a pH of 12 after being treated with NaOCl solution as oxidation capacity of both H_2O_2 and NaOCl solutions would decrease with the increase in the pH.

Table 3: HF membranes mechanical properties

Sample code	Break stress [MPa]		Break strain [%]		Young's Modulus [MPa]	
	Value	SD*	Value	SD	Value	SD
A	3.89	0.073	20.7	2.19	118	2.25
AC3	4.18	0.121	32.2	4.39	114	4.1
AH	4.24	0.085	26.7	2.78	124	5.43
AN	0.82	0.158	2.7	0.523	2.68	0.756

*(SD: Standard deviation)

3.4. Membrane porosity and average pore size

The mean pore size (nm) and porosity of pristine and post-treated membranes are shown in **Table 4**. The results indicated that both NaOCl and H_2O_2 treatment resulted in pore size increase, while a non-significant decrease of pore size was observed in heat-treated samples. No change in membrane porosity was observed after H_2O_2 treatment and thermal treatment, under the selected test conditions, compared to the pristine sample indicating the pore size stability of the treated membranes. However, for PES UF membrane [17], it was demonstrated that H_2O_2 treatment at a high temperature of 100°C and at a treatment time of 60 min led to porosity increases from 52 to 84%. Also, in the case of NaOCl post-treatment samples, porosity % was almost the same.

Table 4: Porosity and pore size distribution of HDHF membranes

Sample	Mean pore radius from BET analysis [nm]	Porosity [%]
A	9.72	80
AC3	8.57	81
AH	12.53	80
AN	11.68	79.4

The above results indicate that the H_2O_2-treated samples have a more uniform surface, less fiber deformation and the highest mechanical strength compared to NaOCl. Accordingly, performance parameters were evaluated for this treatment and compared to pristine and heat-treated samples.

3.5. Membrane performance

3.5.1 Pure water permeability

Permeate flux of pristine and post-treated PS membranes are shown in **Figure 9**. Pure water permeability decreased after H_2O_2 treatment from 21.9 to 11.2 L/m^{2}h.bar, which could be attributed to the variation of pore size on the active layer. Membrane flux is a key parameter in determining the efficiency of the hemodialysis process. Membrane flux directly impactsits ability to remove various molecular weight toxins. Selecting the appropriate membrane type is crucial for optimizing dialysis treatment outcomes and minimizing potential complications.Li et al. [22] treated PES membrane using 5% H_2O_2 for 100 h, at a pH of 9 and 11, which resulted in a non-significant increase of membrane permeability (less than 10 %) due to the stability of the pore structure. A comprehensive study by Ozaya et al. [17] concluded that the highest membrane permeability for treated PES membrane could be obtained using 5 mM H_2O_2 at a higher temperature of 100°C, while, low pore size values can be obtained at lower temperatures and lower concentrations of H_2O_2, which indicates that treating PS based membranes at high temperatures or for prolonged periods result in increasing membrane permeability to some extent. In the current study, mild treatment conditions resulted in a decrease in the membrane permeability. The permeability results for the heat-treated samples showed similar results for the raw sample. Accordingly, urea and creatinine rejection experiments were conducted on both the raw and H_2O_2 treated samples.

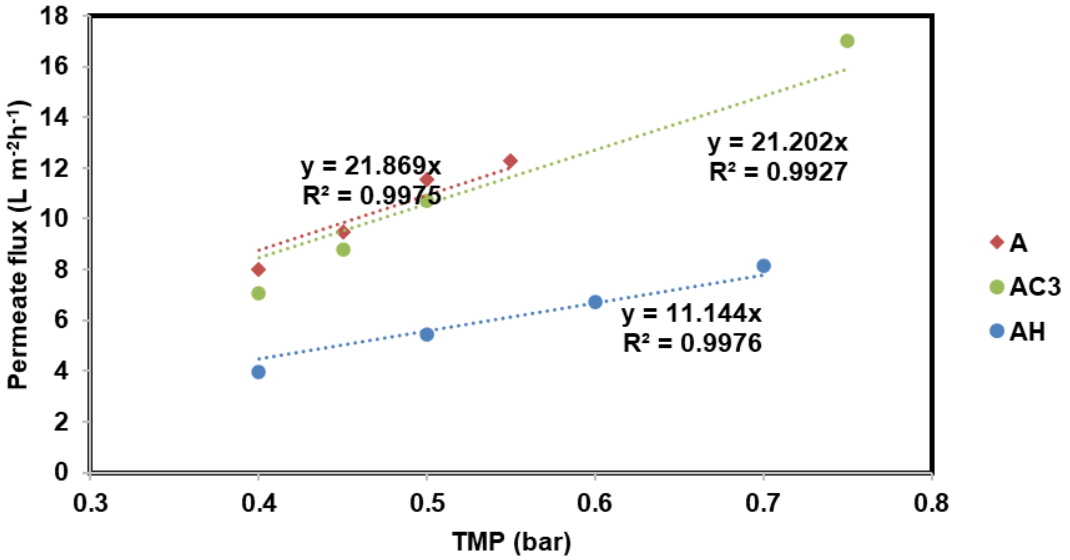


Figure 9: Permeate flux and permeability of pristine and post-treated PS membranes

3.5.2 Sieving coefficient

Urea sieving coefficient approaches unity for the pristine sample and ranged between (0.94 to 0.98) for AH sample after 1 hour filtration. Almost similar results were obtained for creatinine, where the sieving coefficient after 1 hour filtration ranged between (0.98 to 0.996) for sample A, and (0.96 to 0.97) for sample AH as shown in **Table 5**. These results reveal a lower surface pore for H_2O_2post-treated membranes under selected conditions, which were also confirmed by the ultrafiltration coefficient results.

These results are comparable for the prepared raw and H_2O_2post-treated membranes, which agree with previous published results reported by Mansur et al., 2022; Yu et al., 2017 where urea and creatinine sieving coefficients ranged between (0.96-1) and (0.95 to 1), respectively [49,50]. Both raw and H_2O_2 treated samples showed promising membrane performance for application as HD membranes.

Table 5: Sieving coefficient of pristine and H_2O_2post-treated membrane

	Feed Concentration [ppm]	Sieving coefficient (SC)	
		Sample A	Sample AH
Urea	300	1	0.94
	650	1	0.95
	1000	0.99	0.98
Creatinine	60	0.996	0.96
	100	0.988	0.97
	160	0.981	0.97

The effect of ultrafiltration time on the sieving coefficient was also studied. It is seen from **Figure 10**that the sieving coefficient values increase with the increase in the filtration time. In most cases, permeate concentration slightly exceeded feed concentration after three hours of treatment which could be attributed to concentration polarization where urea and creatinine accumulate on the inner surface of the membrane, forming a layer with a higher concentration [51,52].

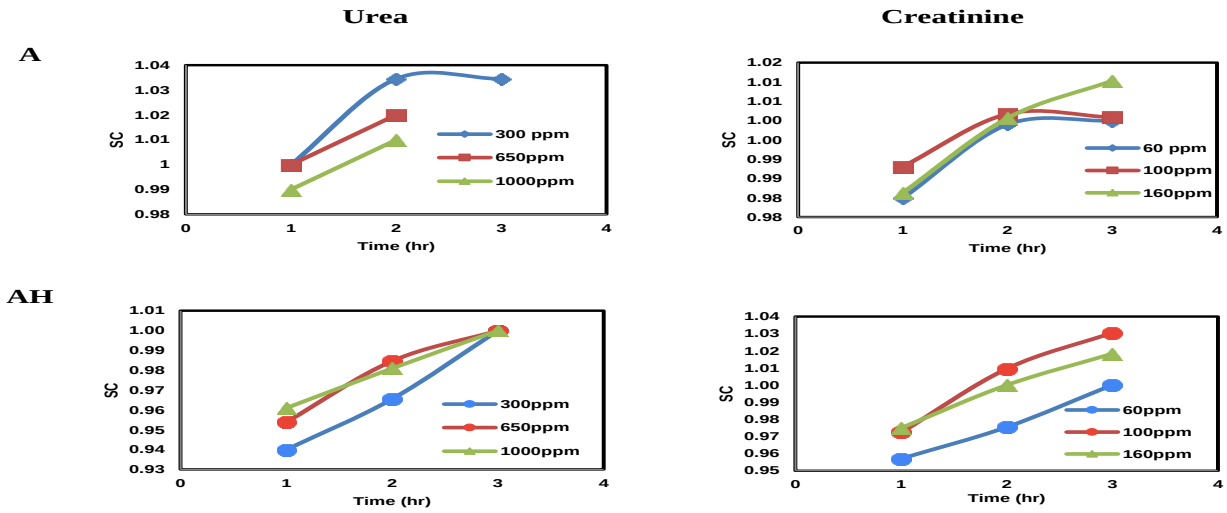


Figure 10: Urea and Creatinine sieving coefficient change over time for pristine and H_2O_2post-treated samples.

Table 6 provides a summary of the impact of NaOCl and H_2O_2 oxidizing agents on the properties of PS and polyether sulfone membranes.

Table 6: Effect of oxidizing agent treatment on membrane characteristics and performance

Membrane type/application	Treatment conditions	Treatment effect on membrane characteristics and performance	Reference
PS/PVP HF/HD	NaOCl (2400ppm) pH 7.5, T=50 1-14 hr.	• Increased surface negativity • Increased pore size • Decreased dextran sieving coefficient • increased UF coefficient	[20]
PS HF/UF	NaOCl 5000 ppm pH 9-9.5, (RT) 2 – 400 hr.	• Increased pore size • Decreased humic acid rejection • Increased CA • Increased surface negativity • Increased water flux	[53]
PS/PVP+PEG HF/UF	NaOCl (50-5000) ppm pH (6.8-11.5), RT 4 hr.	• Pore size and porosity (increased at 50 ppm then decreased at 5000 ppm) • PVP leaching • Increased CA • Increased water permeability	[23]
PES+PVP FS/UF Commercial	H_2O_2 /NaOCl (5000ppm) pH 9-11, 25˚C 6 hr.	• Increased surface negativity • Increased flux	[13]
PES Spiral/UF	NaOCl (700 ppm) pH 9 and 12, 55 C 14-35 days	• Surface pitting and cracking appeared at pH 9 • Decreased tensile and yield strength	[19]
PS+PEG HD	H_2O_2 7500 ppm pH 8. 45˚C 3 hr.	• Increased surface roughness • Increased pore size • Increased break stress and break strain • Insignificant changes in CA • Decreased UF coefficient	This work
PS+PEG HD	NaOCl 7500 ppm pH 8. 45˚C 3 hr.	• Increased surface roughness • Increased pore size • Decreased break stress and break strain • Insignificant changes in CA	This work

FS: Flat sheet, UF: ultrafiltration, HD: hemodialysis

4. Conclusions

This study investigates the effects of selected post-treatment with thermal and oxidizing agents on the characteristics and performance of PS HDHF membrane. Post-treatment with heat or H_2O_2led to a slight reduction in the inner and outer diameters of the membrane fibers, while NaOCl resulted in the thinnest fibers among the treated samples. The membrane contact angle remained stable across all treatments, ranging from 82.43° to 86.8°. Heat and H_2O_2 treatments enhanced the mechanical strength of the membranes, showing increased break strain and stress compared to the untreated fibers. Conversely, NaOCl post-treatment caused significant deterioration, reflected by reduced mechanical strength and altered membrane morphology. Pore sizes increased with NaOCl and H_2O_2 treatments (to 12.5 and 11.7 nm, respectively), whereas heat treatment caused a negligible decrease. Permeability remained unchanged with heat treatment but declined notably after H_2O_2 exposure, dropping from 21.9 to 11.2 $L/m^2 \cdot h \cdot bar$. The urea sieving coefficient remained close to 1.0 in the raw sample and ranged from 0.94 to 0.98 in H_2O_2 treated membranes after one hour of filtration. Overall, the findings suggest that H_2O_2 is a safer and more effective oxidizing agent for modifying hemodialysis PS membranes flux ranges without compromising their physicochemical properties under the studied conditions. Future studies should explore optimizing H_2O_2post-treatment conditions and comparing its performance with other chemical and physical post-treatment to enhance HD membrane efficiency and durability.

Conflicts of Interest

There are no conflicts to declare.

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